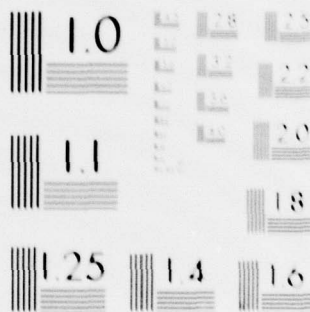


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6 THE OXIDATION OF GRAPHITIC MONOLAYERS ON Ni(110)

by

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ABSTRACT

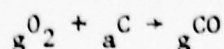
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1. INTRODUCTION

In a previous paper⁽¹⁾ we have described the adsorption and decomposition of ethylene on a clean Ni(110) surface over a wide range of temperatures. At all temperatures studied, the reaction proceeded by dissociative adsorption of the ethylene, with complete dehydrogenation, and desorption of the resulting hydrogen molecules into the gas phase. The surface carbon concentration resulting from this adsorption process was dependent on the temperature at which the adsorption took place. For temperatures below 620K, the final coverage corresponded to one carbon atom per surface nickel atom, probably representing a dehydrogenated C₂ molecular surface species. For temperatures between 620K and 800K, the final coverage corresponded to a monolayer of carbon having an interatomic spacing the same as the C-face of graphite. At higher temperatures no residual carbon was observed on the surface, indicating dissolution into the bulk of the nickel crystal.

We have extended the above study to an investigation of the interaction of these carbonaceous adlayers with gaseous oxygen. This is a reaction that is of interest for at least two reasons. First, it was observed in the previous study that a molecularly adsorbed phase of ethylene was formed on the surface following formation of the dissociatively adsorbed layer at T < 620K. Possible reactivity of this phase with oxygen would be indicated if this surface proved active for the dissociative adsorption of oxygen. Second, the graphitic layer formed at higher temperatures appeared to be inert to further adsorption, and may represent a "poisoned" state of the catalyst surface. If this is so, then reaction of this carbon with gaseous oxygen would provide a mechanism for regeneration of the clean surface. With this in mind, we have observed the kinetics of the overall reaction



over a range of surface temperatures and initial surface carbon concentrations, using Auger electron spectrometry (AES) to measure surface carbon and oxygen concentrations, and a combination of dc mass spectrometric detection and molecular beam relaxation spectroscopy (MBRS) to measure scattered oxygen and product CO signal phase and amplitude. In the present report we discuss the results obtained at $T > 700\text{K}$. The results for $T < 700\text{K}$ will be presented separately.

2. EXPERIMENTAL

The measurements were carried out in the surface research system used for the previous studies, and described in detail in the report of that work.⁽¹⁾ In brief, it consists of a large metal ultrahigh vacuum chamber. The surface under study is mounted on the axis of the chamber using a universal positioning device. The reactant beam is formed by a supersonic nozzle source in a differentially-pumped auxiliary chamber, and modulated in a second differentially-pumped chamber before it enters the main reaction chamber. The main chamber also contains the mass spectrometer used to detect the scattered product signal, a cylindrical mirror Auger electron spectrometer, and an ion gun for surface cleaning between experimental runs.

Two modifications have been made to this system since the previous study. The detector mass spectrometer has been fitted with a new ion source, which has resulted in an improvement of the signal-to-noise ratio of the detector by about a factor of five. The original beam modulation system, a large, multi-toothed chopper wheel driven by a motor external to the vacuum system, has been replaced by a much smaller wheel, driven by a motor inside the collimator chamber. Installation of this system has increased the available modulation frequency range to 15-300 Hz. In the present work the mass spectrometer detector output was sensed by a Princeton Applied Research

Model 2504 two-phase lock-in amplifier. This unit permits simultaneous measurement of product signal phase and amplitude, and has proven invaluable in following the rapidly-changing CO product signal phase observed during the surface oxidation reaction at high temperatures.

The sample used in this study was the same one used in the previous work,⁽¹⁾ a nickel single crystal 2.5 cm long, 0.6 cm wide and 0.025 cm thick, with a (110) orientation on the flat surface. It was cleaned prior to the experimental studies and between runs by cycles of argon ion bombardment followed by anneal at 800K, until the Auger spectrum of the crystal showed only those peaks typical of the clean nickel surface. In this phase of the work, carbonaceous layers were formed on the nickel surface by exposure to an ethylene molecular beam at temperatures above 600K until saturation of the carbon AES signal was observed.

This process was observed in the previous work⁽¹⁾ to lead to decomposition of the ethylene, with desorption of the hydrogen as H₂ and formation of an adlayer structure having a surface carbon atom concentration of 3.35×10^{15} carbon atom/cm². This concentration is essentially the same as the number of carbon atoms per cm² in the C-face of graphite. The carbon AES peak associated with this structure had the typical "graphitic" shape. We thus refer to this structure as a "graphitic monolayer".

Two techniques were used to follow surface reaction kinetics for all experimental conditions studied. Auger spectroscopy was used to monitor surface carbon and oxygen concentrations as exposure to ethylene or oxygen took place. Previous studies have indicated that in this case the presence of the Auger primary electron beam does not observably affect the kinetics of the surface processes involved. The flux of molecules from the surface during the reaction process was measured mass spectrometrically, using a modulated reactant beam and the two-phase lock-in amplifier at the mass

spectrometer output to obtain information on both the magnitude and phase of the scattered reactant and product species signals.

3. RESULTS AND INTERPRETATION

For the case to be discussed here, the reaction of oxygen with the graphitic monolayer at temperatures above 700K, the observed gas-phase reaction product was in all cases CO. The kinetics of the oxidation reaction at these high temperatures are complicated by a number of competing reactions. Those of particular importance are the dissolution of the carbon layer in the bulk, the dissolution of adsorbed oxygen into the bulk, and the reaction between oxygen and dissolved carbon in the late stage of the reaction sequence.

In a separate study⁽²⁾ we have determined that, in the absence of oxygen, the carbon dissolution reaction is limited by the rate of removal of carbon atoms from the monolayer structure. This reaction is slow compared to the bulk diffusion rate of carbon at all temperatures. At temperatures below 870K it is also slow compared to the observed rate of the oxidation reaction. Thus oxidation measurements in this temperature range are only slightly affected by the dissolution reaction.

The overall course of the oxidation reaction in this temperature range has been characterized by Auger spectrometric measurement of surface carbon and oxygen concentrations, and by a.c. mass spectrometric measurement of reflected oxygen and product CO signal phase and amplitude. The results of a series of measurements at 853K are shown in Figure 1.

Consideration of the data presented in this figure reveals much qualitative and quantitative information about the course of the surface reactions involved. The reaction to form CO is initially very slow, followed by a region of rapid reaction, with the reaction rate again decreasing as the carbon supply is exhausted. This is shown both by the

curve of carbon AES signal with time, and by the product CO mass spectrometer signal. The relationship between these two signals is shown quantitatively in Figure 2. Here we have integrated the CO mass spectrometer signal, normalized the integrated value to the total amount of carbon present in the layer initially and plot $(1-f_{CO})$ vs. time for comparison with the carbon AES signal. There is quantitative correspondence between carbon disappearance and CO evolution down to a carbon coverage, θ_c , of about 0.3. Beyond this point CO evolution exceeds the carbon removal rate, probably due to reaction of surface oxygen with carbon dissolved in the near-surface bulk in the earlier stages of the reaction.

The oxygen adsorption rate appears to be limited to surface area not covered by the carbon monolayer, as the oxygen mass spectrometer signal, which is a measure of the fraction of the oxygen flux that does not chemisorb, follows the carbon AES signal very closely until late in the reaction sequence, as can be seen in Figure 1.

In the early stage of the reaction, the total oxygen uptake, as determined by graphical integration of the area between the observed oxygen scattering curve and the maximum oxygen signal line, is equal to the amount of carbon removed. This relation is also shown in Figure 2, where the oxygen uptake is plotted as $(1-f_{O_2})$ vs. time. This relationship holds down to a θ_c of about 0.8. After this point the oxygen uptake exceeds the carbon removal. Since no oxygen is observed on the surface by AES until much later in the reaction, the additional oxygen taken up must be dissolving in the bulk of the nickel. A separate experiment, in which the clean nickel surface was exposed to oxygen at 853K, indicated rapid dissolution of adsorbed oxygen, as indicated by disappearance of the AES signal when the oxygen beam was turned off. An oxygen adlayer containing $\sim 4 \times 10^{15}$ atom/cm² disappeared completely in 140 seconds.

The data described above are sufficient to permit a quantitative description of the surface reaction process. The initial very slow rate, and the correspondence between the oxygen uptake rate and carbon removal rate at short times suggest a mechanism wherein oxygen adsorbs only at defects initially present in or nucleated in the film, and that the oxygen adatoms, once formed, react rapidly and quantitatively with carbon atoms adjacent to the defects. The reaction thus proceeds by growth of holes in the film around the nucleation sites. At later stages in the reaction, the radius of the holes in the film increases to the point where dissolution of adatoms in the bulk is of comparable probability with reaction at hole perimeter sites. Further reaction then proceeds at a rate limited by oxygen adsorption within a surface diffusive mean free path of the hole perimeter.

We have evaluated the appropriate kinetic equations for these two cases in the Appendix to this Paper. The resulting expression for the short-time case is

$$\theta_c = \exp(-a_0 N_0 e^{Kt})$$

in which θ_c is the fraction of the initial carbon layer remaining at time t , a_0 the area of an initial defect or hole nucleation site, N_0 the number of nucleation sites per unit area and K is given by

$$K = 2S_0 J A,$$

in which $2S_0 J$ is the oxygen uptake rate per unit area of available surface and A is the area per carbon atom in the film.

For the experimental conditions used here, S_0 , the sticking coefficient for chemisorption on the clean surface is observed to be 0.8, J , the oxygen flux, was measured to be 2.7×10^{13} molec/cm²sec. The area per carbon atom in the film is 3.0×10^{-16} cm². This leads to $K = 1.3 \times 10^{-2}$ sec⁻¹.

The values of a_o and N_o cannot be obtained from direct measurement. Their product thus represents an adjustable parameter in fitting the kinetic expression to the experimental data. We find that the value $a_o N_o = 7.3 \times 10^{-5}$ gives a very good fit to the experimental data in the range $1.0 \geq \theta_c \geq 0.8$ as shown in Figure 3. If we assume that the critical size required for a defect to be an effective adsorption site for oxygen is roughly the area occupied by two nickel atoms, i.e., $9 \times 10^{-16} \text{ cm}^2$, this yields a value of $N_o = 8 \times 10^{10} \text{ cm}^{-2}$, or roughly one atomic site in 10^4 . This is felt to be a reasonable value, as it is of comparable magnitude to values deduced by similar arguments for other systems. (3,4)

We have also been able to fit that part of the experimental curve beyond $\theta_c = 0.8$ with the model developed in the Appendix for the case in which only oxygen adatoms formed within a finite distance from the hole perimeter contribute to the oxidation reaction. For this case the kinetic equation developed is

$$\theta_c = \exp(-K't^2),$$

in which $K' = \pi(4K\bar{x})^2 N_o$

where \bar{x} is the effective limiting distance over which an oxygen adatom can diffuse to the hole perimeter without being lost by dissolution into the bulk. In fitting this relation to the experimental data it is necessary to assume a fictitious $t_o < t$ in order to match the initial portion of the carbon AES curve at $\theta_c = 0.8$. The best fit between the kinetic expression and the data was obtained for $t_o = 260 \text{ sec}$. $K' = 4.3 \times 10^{-6} \text{ sec}^{-2}$, and from this, making the same assumptions as before, $\bar{x} = 2 \times 10^{-7} \text{ cm}$, a physically reasonable value.

The amplitude and phase of the CO product signal observed mass spectrometrically are also in accord with the model developed. The phase of the CO signal, shown plotted in Figure 1 as phase delay relative to the scattered unreacted oxygen signal, starts at a low value, rises, and then levels off at a constant value. The constant phase region is reached at approximately the same value of θ_c as the postulated changeover in reaction mechanism. This is as expected, as during the initial stage of the reaction, the mean distance travelled by an oxygen adatom prior to reaction to form CO is increasing with time and consequently the time elapsed between adsorption and reaction is increasing. In the later stages of the reaction, when only those oxygen adatoms formed within \bar{x} of the hole perimeter contribute to the reaction, the mean distance travelled, and hence the time involved in the transport process, is constant. The integrated amplitude of the CO signal also provides quantitative agreement with the mechanism postulated. If all of the carbon initially present in the film is removed as CO, then the integral of the area under the CO signal curve up to any time should be equal to the amount of carbon removed, as shown by the carbon AES signal. It can be seen from Figure 2 that this is the observed behavior, as the integral curve matches the carbon AES curve within experimental error down to $\theta_c = 0.3$. At this point the integral indicates somewhat less carbon removal. This discrepancy is thought to be due to the reaction between oxygen and carbon that has been dissolved in the bulk during earlier stages of the reaction.

It is thus seen that there is internal consistency among all of the experimental measurements, and essentially quantitative agreement between these measurements and the model developed to account for the experimental results. The degree of agreement, and the wide variety of independent measurements made lead to a high degree of confidence in the validity of the proposed mechanism.

SUMMARY

The results presented demonstrate that even in such an apparently simple surface reaction as the oxidation of carbon to CO, a wide range of kinetic processes are active. In order to understand and to characterize these kinetic processes, a wide range of experimental techniques must be brought to bear. The combination of AES and molecular beam mass spectrometry has enabled us to sort out the complex reaction sequence and to obtain a quantitative description of the surface processes taking place.

The picture of a reaction rate limited at short time by the availability of sites for oxygen adsorption and at long times by oxygen surface diffusivity emerges clearly from the results obtained from the simultaneous application of multiple surface probes. The results presented provide the best quantitative explanation that we have obtained to date on the course and mechanism of a surface chemical reaction. The quantitative agreement among the carbon AES data, the oxygen uptake, as measured mass spectrometrically, and the CO evolution rate leads to a high degree of confidence in the theoretical model developed to fit the overall reaction rate. In spite of the complications associated with competing carbon and oxygen dissolution processes, the picture of a reaction that proceeds by the nucleation and growth of holes in the graphitic layer emerges convincingly.

ACKNOWLEDGMENT

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APPENDIX

We wish to develop reaction rate equations for the case of a reaction limited by the availability of adsorption sites for the gas-phase species at short times, and by capture of adsorbed atoms at the perimeter of holes in the initial surface film at long times, where the hole radius is large compared to the mean free path for adatom surface diffusion.

For either case, the overall extent of reaction, in this case the fraction of the surface carbon layer removed in a given time, θ , can be expressed by an equation developed by Holloway and Hudson⁽³⁾ as

$$\theta = 1 - \theta_c = 1 - \exp\left(-\int_0^t a_\eta i d\eta\right), \quad (\text{A-1})$$

in which θ_c is the fraction of the initial carbon layer remaining at time t , a_η is the area of a hole in the film nucleated at time η and i the nucleation rate of holes. This expression resulted from extending the treatment of bulk-phase precipitation developed by Johnson and Mehl⁽⁵⁾ and by Avrami⁽⁶⁾ to the case of two-dimensional symmetry. Since what is measured in the Auger spectroscopic study of the oxygen reaction is θ_c , we will rewrite the above to yield

$$\theta_c = \exp\left(-\int_0^t a_\eta i d\eta\right). \quad (\text{A-2})$$

It remains now to develop expressions for a_η and i appropriate to the cases of interest. Consider first the short-time case. In this portion of the reaction sequence, essentially all of the oxygen that is chemisorbed is used in the reaction to form CO. The rate of the process is limited only by the adsorption rate, which in turn is limited by the availability of nickel surface sites not covered by the carbon layer. For this case we may write

$$a_{\eta} = \int_{a_0}^a da = \int_{\eta}^t \left(\frac{da}{dt} \right) dt, \quad (A-3)$$

where $\frac{da}{dt}$, the growth rate of the island, is given by

$$\frac{da}{dt} = 2 S_o J A a, \quad (A-4)$$

in which S_o is the oxygen sticking coefficient in the clean nickel surface, J the oxygen flux to the surface and A the area per surface carbon atom. Insertion of this value into A-3 and integration leads to

$$a_{\eta} = a_0 \exp [2 S_o J A (t-\eta)]. \quad (A-5)$$

The nucleation rate of islands can be treated identically to the case previously considered by Holloway and Rudson,⁽³⁾ assuming that the holes in the film nucleate in a time short compared to the time of the experiment. If at $t=0$, N_o potential nucleation sites exist, if the nucleation probability per unit time per site is v , and if after time η , N nucleation sites remain, then the nucleation rate $i = dN/dt$ is given by

$$i = N_o v \exp(-v\eta). \quad (A-6)$$

Substituting A-5 and A-6 into A-2 yields

$$\theta_c = \exp \left\{ - \int_0^t a_0 \exp[K (t-\eta)] N_o v \exp(-v\eta) d\eta \right\} \quad (A-7)$$

in which

$$K = 2 S_o J A. \quad (A-8)$$

Integration yields

$$\theta_c = \exp \left[- \frac{a_0 N_o v}{K+v} (e^{-vt} - e^{-Kt}) \right], \quad (A-9)$$

If the nucleation process is rapid compared to the growth process, then at appreciable values of t , we will have $e^{Kt} \gg e^{-vt}$. Moreover, we will also have $v \gg K$. Making these assumptions leads to

$$\theta_c = \exp [-a_o N_o e^{Kt}]. \quad (A-10)$$

This is the final expression that we will use to describe the reaction rate at short times.

Later in the reaction sequence, as the holes in the film grow, a point is reached where there is competition for adsorbed oxygen atoms between the CO-production reaction and dissolution of oxygen atoms into the bulk of the nickel. Once this situation has developed, only those oxygen adatoms that are formed within a finite distance from the hole perimeter, \bar{x} , will, on the average, reach the perimeter and react to form CO. For this case, it is more convenient to work with r_η , defined by $a_\eta = \pi r_\eta^2$. Thus,

$$r_\eta = \int_0^r dr = \int_\eta^t \left(\frac{dr}{dt}\right) dt. \quad (A-11)$$

For this case, using the random walk distance⁽⁷⁾ to determine the number of molecules reaching a perimeter site per second, $\frac{dr}{dt}$ is given by:

$$\frac{dr}{dt} = 8A S_o J \bar{x} \quad (A-12)$$

Insertion of this value into A-11 and integration leads to

$$r_\eta = 8A S_o J \bar{x} (t-\eta) \quad (A-13)$$

or

$$a_\eta = \pi (8A S_o J \bar{x})^2 (t-\eta)^2 \quad (A-14)$$

If we make the same assumptions about the nucleation process as in the previous case, we arrive at

$$\theta_c = \exp \left\{ - \int_0^t \pi (8A S_o J \bar{x})^2 (t-\eta)^2 N_o v \exp(-v\eta) d\eta \right\}, \quad (A-15)$$

which can be rearranged to yield

$$\theta_c = \exp \left\{ - \pi (8A S_o J \bar{x})^2 N_o v \int_0^t (t-\eta)^2 \exp(-v\eta) d\eta \right\} \quad (A-16)$$

Integration by parts, and the previous assumption that vt is very large, leads to

$$\theta_c = \exp \left(- \pi (8A S_o J \bar{x})^2 N_o t^2 \right), \quad (A-17)$$

or, if we lump all constant terms as

$$K' = \pi (8A S_o J \bar{x})^2 N_o = \pi (4K\bar{x})^2 N_o \quad (A-18)$$

we have

$$\theta_c = \exp (-K' t^2) \quad (A-19)$$

The final expression that we will use to describe the reaction rate at long times.

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FIGURE CAPTIONS

- Figure 1: Summary of kinetic measurements of the oxidation of a graphitic carbon monolayer on Ni(110). (o) carbon coverage, by AES; (\square) amplitude of scattered O_2 mass spectrometer signal; (\blacklozenge) oxygen coverage, by AES; (\bullet) amplitude and (Δ) phase of product CO mass spectrometer signal.
- Figure 2: Comparison of CO production and total O_2 uptake with carbon removal in the oxidation of a graphitic carbon monolayer on Ni(110). (o) carbon coverage, by AES; — CO produced; --- O_2 uptake.
- Figure 3: Comparison of theoretical predictions to the observed extent of reaction in the oxidation of a graphitic monolayer on Ni(110). (o) carbon coverage by AES.

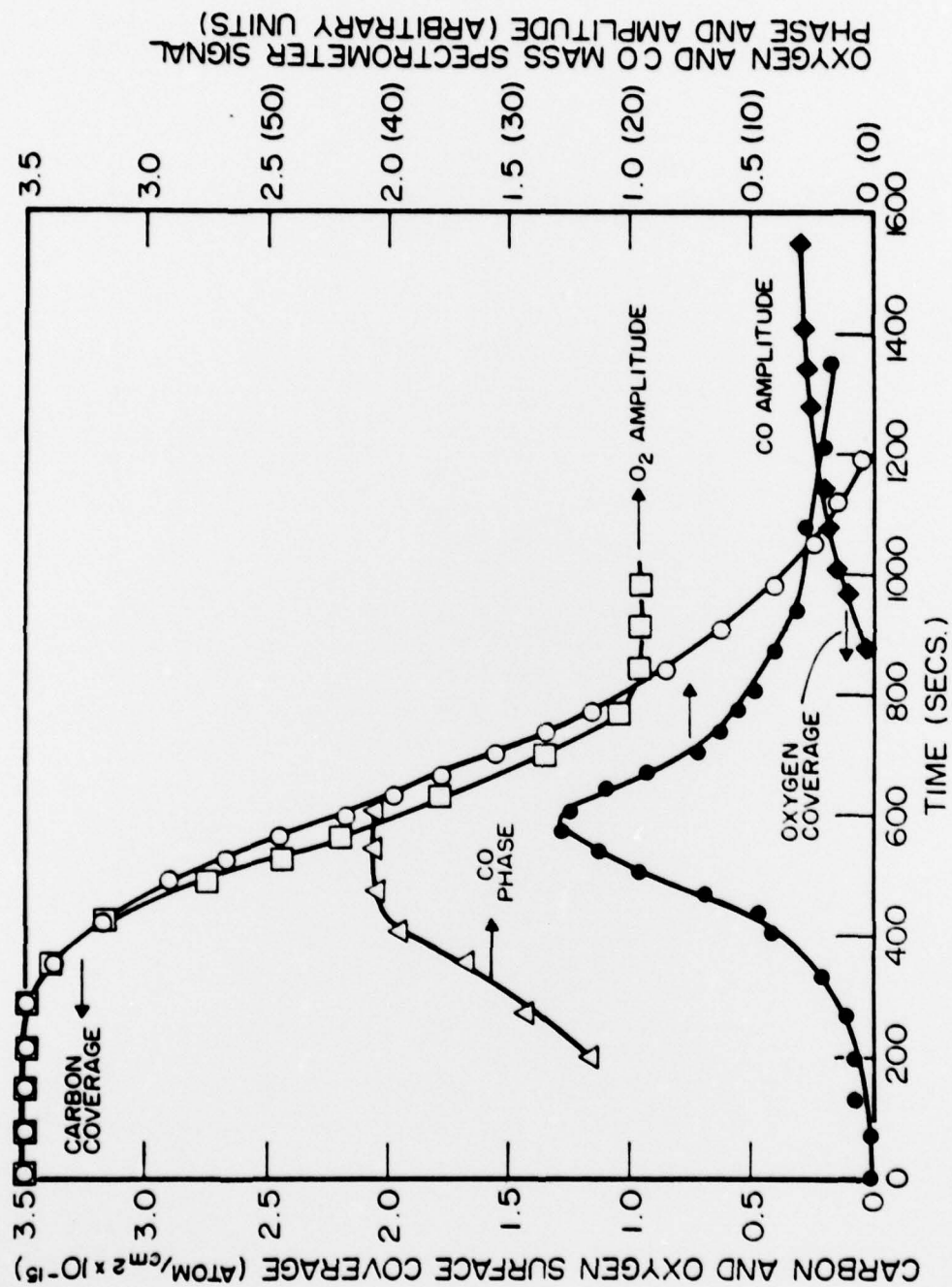


Figure 1

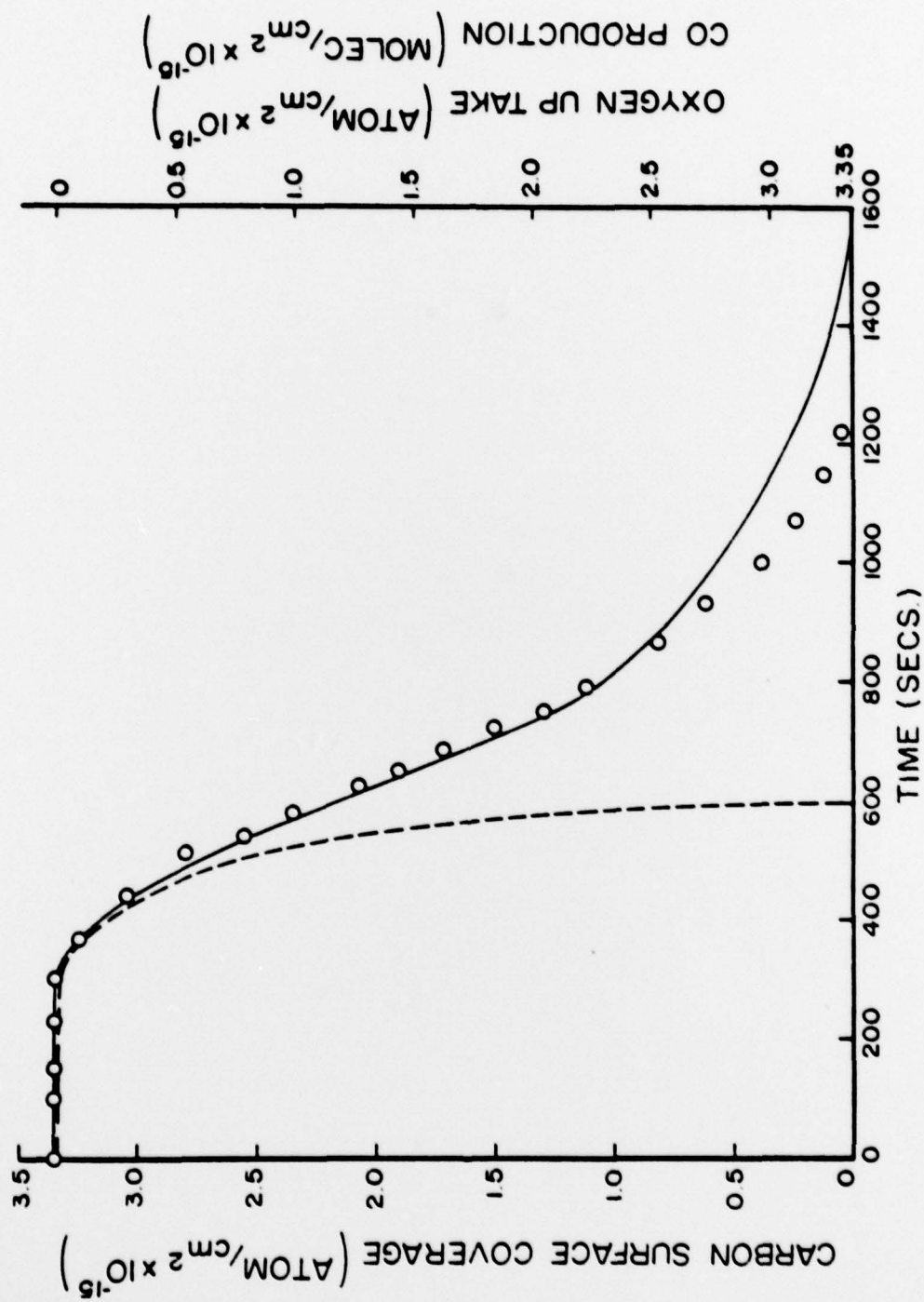


Figure 2

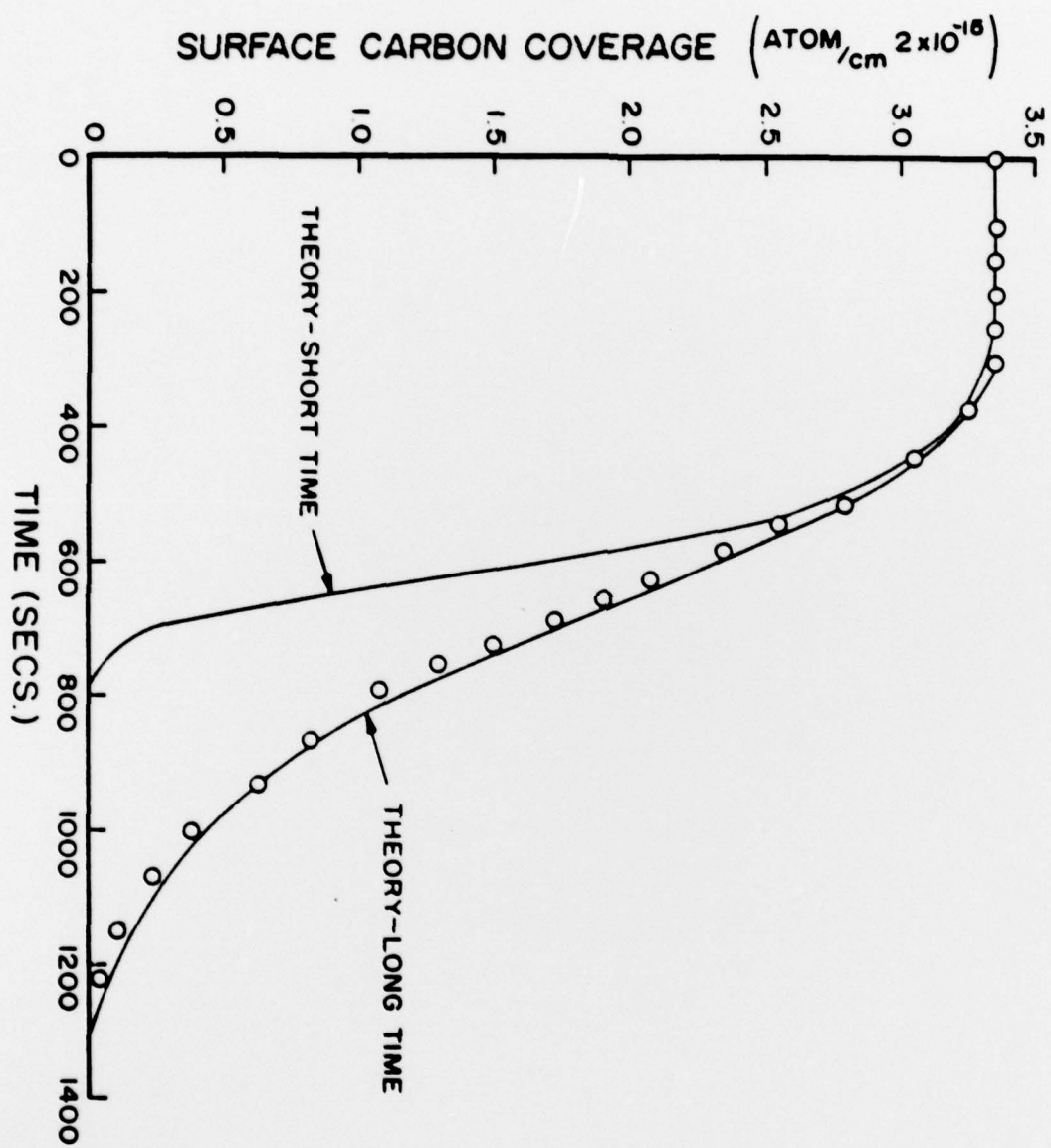


Figure 3